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Microstructural Control of a Precipitate-Hardenable Al-Ag Alloy Using Severe Plastic Deformation

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Abstract. An Al-10.8wt%Ag alloy was subjected to aging treatment followed by Equal-Channel Angular Pressing (ECAP) (designated process AE) or ECAP followed by aging treatment (designated process EA). Hardness measurements were undertaken with respect to the number of ECAP passes for process AE or with respect to aging time for process EA. Microstructures were examined by transmission electron microscopy (TEM) including X-ray mapping. It is shown that age hardening is observed for the ECAP sample due to the precipitation of very fine particles within the small grains.

Introduction

It is well known that the process of equal-channel angular pressing (ECAP) is used to reduce the grain size of metallic materials to the submicrometer range or the nanometer range [1-3]. However, it is not so well established that the ECAP process may also be used to control the morphology and distribution of second phase particles in two-phase metallic materials. Thus, grain refinement and second-phase control are both feasible because severe strain is created through the process of ECAP based on the principle that a sample is pressed through a channel bent into an L-shape within a die and this pressing may be repeated without any change in the cross-section of the sample. Fragmentation not only of the grains but also of second-phase particles may occur due to the introduction of severe strain in the material.

Although many reports have now been published for grain refinement using the ECAP process [2,3], there are only a limited number of examples of the application of the ECAP process to second-phase control [4,5]. It was shown that the application of ECAP led to dissolution of θ' particles precipitated by aging of an Al-3.7wt%Cu alloy [4]. Supersaturation thus occurred in the alloy and subsequent aging gave rise to the formation of a stable θ–phase. The θ–phase precipitation was also recognized after aging of a severely deformed Al-3.7wt%Cu alloy using ECAP. An application of ECAP to an Al-0.9wt%Mg_2Si alloy resulted in microstructural changes similar to the Al-3.7wt%Cu alloy except that cube-shaped particles were formed following aging of the alloy in the supersaturation due to dissolution by ECAP [5]. Although the cubed-shaped particles remained small after prolonged aging, there have been no successful increases in the strength of the alloys compared with normal aging treatments.

Aluminum alloys containing Ag are also well known to exhibit hardening by aging. The aging behavior including micostructural evolution is similar to Al-Cu and Al-Mg_2Si alloys with the formation of GP zones through a metastable phase to a stable phase [6]. This study was therefore initiated to apply ECAP to second-phase control in an Al-Ag alloy. Transmission electron microscopy (TEM) was used to examine microstructural changes associated with ECAP and aging.

Experimental Procedures

An Al-10.8wt%Ag alloy was prepared from high purity (99.99%) Al and high purity (99.9%) Ag. An ingot of the alloy was produced using a melting and casting procedure and homogenized at 753 K for 24 hour. The ingot was swaged at room temperature to the form of rods with diameters of 10 mm and they were cut into lengths of 60 mm. These rods were solution-treated at 823 K for 1 hour, followed by quenching into iced water. The processes of aging and ECAP were carried out one after the other. In process AE, aging was first undertaken at a temperature of 473 K for periods of up to 300 hours and ECAP was then conducted for the samples aged for 10 hours. In process EA, ECAP was first conducted for 8 passes after solution treatment and then the ECAP samples were subjected to aging at temperatures of 373 or 473 K for periods of up to 300 hours. In both processes, the ECAP was conducted at room temperature using a die having a channel angle of 90°, which creates an equivalent strain of ~1 for one passage through the die [7]. The sample was rotated by 90° about the longitudinal axis in the same sense between consecutive passes where this is generally designated processing route B_C [8]. Microstructures were examined using conventional transmission electron microscopy (H-8100, 200 kV) and analytical electron microscopy (TECNAI-20, 200 kV and JEM-2010FEF, 200 kV). The Vickers microhardness was measured for the samples subjected to ECAP and/or aging in both process AE and process EA. The average values were obtained from ten measurements on each sample.

Results and Discussion

Process AE (aging followed by ECAP)

Figure 1 plots the Vickers microhardness against the number of ECAP passes for the samples aged for 10 hours at 473 K. This aging time gave the maximum hardness for the aging treatment. The hardness continuously increases with increasing number of ECAP passes. This behavior is different from earlier results obtained with Al-3.7wt%Cu and Al-0.9wt%Mg_2Si alloys where the hardness reached maxima but decreased with further pressings [4,5].

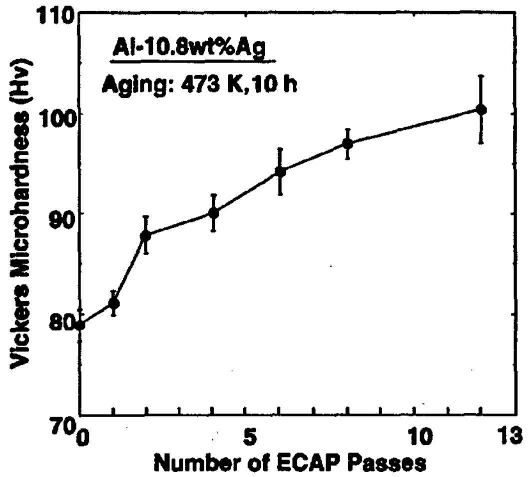

Fig.1 Vickers microhardness against number of ECAP passes for samples aged for 10 hours at 473 K.

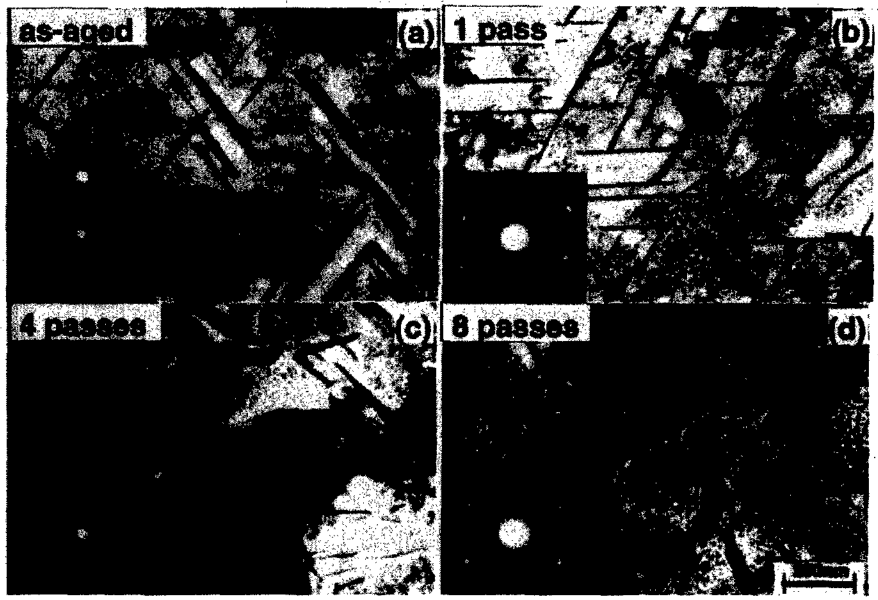

Fig.2 TEM bright field images with SAED patterns: (a) after aging for 10 hours at 473 K and after (b) 1 pass, (c) 4 passes and (d) 8 passes of ECAP following aging for 10 hours at 473 K.

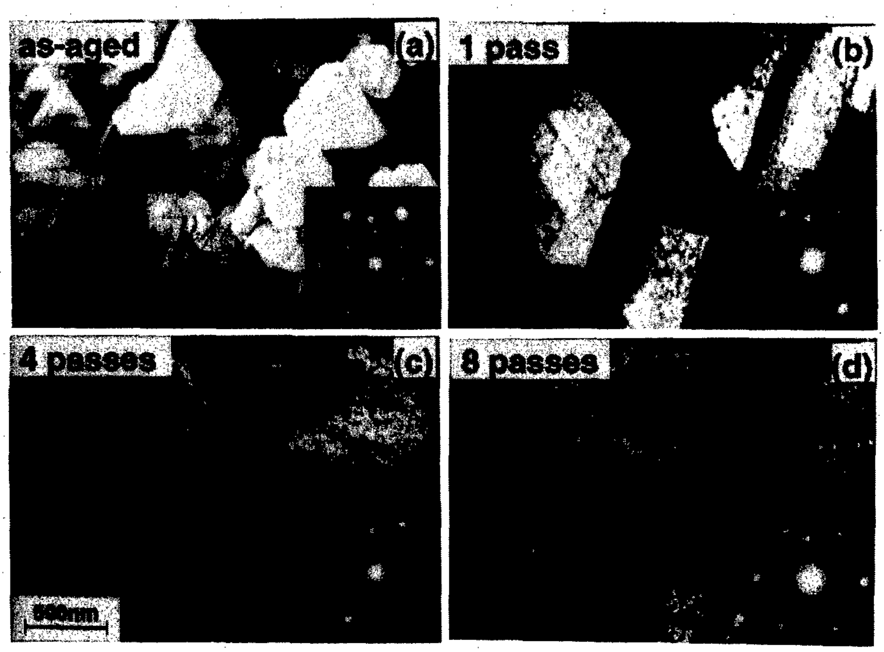

Fig.3 TEM dark field images with SAED patterns: (a) after aging for 10 hours at 473 K and after (b) 1 pass, (c) 4 passes and (d) 8 passes of ECAP following aging for 10 hours at 473 K.

Figure 2 shows TEM bright field images together with selected area electron diffraction (SAED) patterns (a) after aging for 10 hours at 473 K but before ECAP and (b) after 1 pass, (c) after 4 passes and (d) after 8 passes of ECAP following the same aging treatment. All images were taken from the [110] orientation of the matrix. The precipitation of a second phase occurred in the matrix and these precipitates correspond to a metastable γ'-phase as they are known to form in the direction parallel to the matrix {111} planes [9]. These γ'-phase precipitates are distorted after 1 pass of ECAP and the distortion becomes more pronounced increasing the number of ECAP passes.

Figure 3 shows TEM dark field images with SAED patterns. The samples were aged for 10 hours at 473 K and they are (a) before ECAP, (b) after 1 pass, (c) after 4 passes and (d) after 8 passes. The images were taken from the [111] orientation of the matrix using diffracted beams indicated by arrows. In Fig.3 (a), the γ'-precipitates appear extended over certain areas and this is consistent with a report that the precipitation of the γ'-phase occurs in the form of a plate. It is known that the γ'-phase has an hexagonal crystal structure and the plates are formed with the (0001) plane parallel to the {111} planes of the matrix [9]. The brightness of the γ'-phase decreases with increasing numbers of ECAP passes. This observation indicates that the plates tend to be more out of the matrix {111} plane with increasing numbers of passes and this is consistent with the heavy distortion observed in Fig.2.

Figure 4 shows (a) an annular dark field (ADF) image and the corresponding X-ray maps with (b) the Al K_α line and (b) the Ag L_α line, where all image and maps were taken in the scanning transmission electron microscopy (STEM) mode. The sample was aged for 10 hours at 473 K followed by 4 passes of ECAP. The γ'-precipitates exhibit bright contrast in Fig.4 (a) and Fig.4 (c) because they are rich in Ag and dark contrast in Fig.4 (b) because they are depleted in Ag. It is again demonstrated that the precipitates were heavily deformed by ECAP and, as indicated by arrows in Fig.4 (a) and (c), there is clear evidence of an intense shear dividing the precipitate into two parts. A close inspection of Fig.4 (c) further reveals that there are regions of Ag depletion around the precipitates. Such an Ag depletion was observed for the as-aged sample [9] and it seems that there is less effect of the severe deformation due to ECAP on the presence of the Ag-depleted zone near the precipitates. It seems also that the severe deformation does not create dissolution of Ag into the matrix unlike the cases observed in Al-3.7wt%Cu and Al-0.9wt%Mg_2Si alloys.

Fig.4 (a) Annular dark field (ADF) image, (b) Al K_α line map and (b) Ag L_α line map for sample aged for 10 hours at 473 K followed by 4 passes of ECAP.

Process EA (ECAP followed by aging)

Figure 5 plots the Vickers microhardness against the aging time for the samples subjected to 8 passes of ECAP. The hardness variation is also shown for the solution-treated samples without ECAP. The hardness increases with aging for the as-solution treated samples and exhibits maxima for both aging temperatures but with a rate which is faster at 473 K than at 373 K: the maximum hardness was obtained after aging for 10 hours at 473 K while it was after 100 hours at 373 K. However, the aging behavior is very different for the ECAP samples. The hardness continuously

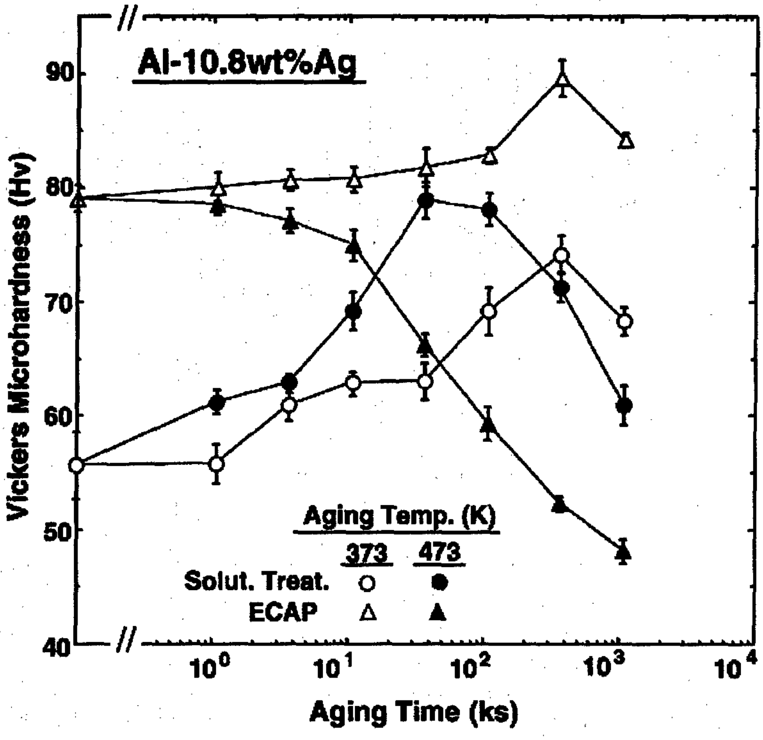

Fig.5 Vickers micro-hardness against aging time for samples subjected to 8 passes of ECAP and for solution-treated samples without ECAP.

decreases from the initial stage with aging at 473 K, but it gradually increases with aging at 373 K and reaches a maximum after aging for 100 hours which is the same aging time as for the as-solution-treated sample. The maximum hardness obtained by aging the ECAP sample at 373 K is well above the maximum hardness for the as-solution-treated samples.

Observations by TEM were conducted for the ECAP sample aged at 373 K for 100 hours leading to the maximum hardness and a microstructure is shown in Fig.6 (a). For comparison, a microstructure is also shown in Fig.6 (b) from the ECAP sample aged at 473 K for the same aging period. It is confirmed that there is a significant difference in the microstructures. Three representative features are noted in association with the microstructure shown in Fig.6 (a): (1) small grains having sizes of less than 1 μm but containing dislocations with the grains as marked A, (2) very small particles which are finely dispersed within the grains as marked B, and (3) some large particles which are mostly present on the grain boundaries as marked C. The fine-grained structure with a high dislocation density is a typical microstructure observed in ECAP samples [10]. A preliminary observation using high-resolution analytical electron microscopy showed that the very fine particles formed within the grains may be a GP zone equivalent to the η–zone. The large particles are considered to be the stable γ–phase as they are not coherent with the matrix. By contrast, the microstructure after aging the ECAP sample at 473 K for 100 hours contains neither small grains nor very small particles within the grains but large γ-phase particles are present. It is considered that the age hardening observed in the sample aged at 373 K is attributed to the formation of the very small particles within the grains and the hardness remains the highest because of the simultaneous effect of the grain refinement due to the ECAP process and the hardening due to the low temperature aging which does not lead to grain growth.

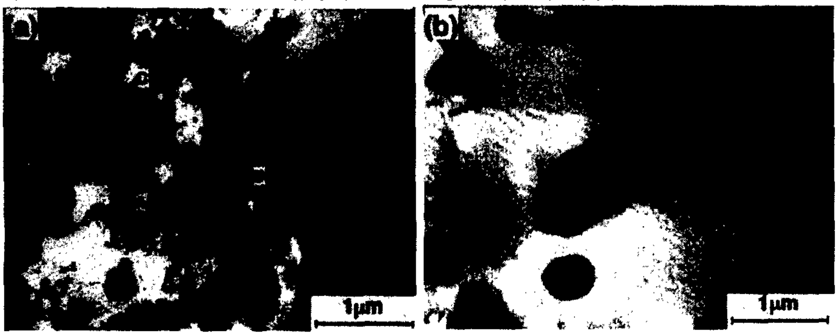

Fig.6 TEM micrographs for ECAP samples aged for 100 hours at (a) 373 K and (b) 473 K.

Summary and Conclusions

1. An Al-10.8wt%Ag alloy was subjected to aging treatment at 473 K for 10 hours followed by ECAP up to12 passes (Process AE) or to 8 passes of ECAP followed by aging treatment at 373 K and 473 K for periods of up to 300 hours (Process EA).
2. For process AE, the hardness continuously increased with increasing numbers of ECAP passes. The metastable γ'-phase in the form of plate-like precipitates was heavily deformed by ECAP. No dissolution of Ag into the matrix occurred but Ag-depleted zones remained present after severe deformation through ECAP.
3. For process EA, the hardness continuously decreased with aging at 473 K but it gradually increased with aging at 373 K and reached a maximum after 100 hours. Observations by TEM revealed that small grain sizes less than 1 μm were retained and very small particles were precipitated within the grains after the aging at 373 K for 100 hours, whereas large stable γ–phase particles were present in the coarse-grained matrix after the aging at 473 K for 100 hours.
4. The precipitation of very small particles within the fine-grained matrix obtained in process EA is responsible for the age hardening.

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